

UDC 666.263.2:666.113.621'34'28

IMPROVEMENT OF LITHIUM-ALUMINOSILICATES GLASS COMPOSITION FOR PRODUCTION OF HIGH-STRENGTH GLASS CERAMICS

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Translated from Steklo i Keramika, No. 2, pp. 10–11, February, 2004.

Lithium-aluminosilicate glasses have been melted and the physicochemical properties of glasses and glass ceramics crystallizing in two stages on the basis of these glasses have been studied. The study results are discussed.

Production of glass ceramics involves a wide range of problems related to the nature of glass and variety of physicochemical processes taking place in transformation of glass into glass ceramics. At the same time, problems arising in development of glass ceramic technologies in some cases call for implementation of totally new techniques.

Numerous papers have been published regarding glasses based on the lithium-aluminosilicate system [1] and practical studies regarding glass ceramics [2]. A combination of such properties as high mechanical strength, hardness, abrasion resistance, and high chemical resistance makes glass ceramics promising for extensive applications (civil engineering, medicine, military equipment, etc.). There are glass ceramics with higher strength parameters, for instance, cordierite materials; however, they require a higher temperature for melting initial glasses. In contrast, glasses of the lithium-aluminosilicate system are lower-melting, which allows for saving energy in their production and does not require expensive refractories.

The purpose of our study is modification and improvement of known lithium-aluminosilicate glass compositions for production of high-strength glass ceramics (Table 1).

Four glasses based on the formulas indicated in RF Patent No. 216712 were synthesized and analyzed. The weight content of lithium in this glasses varies within a range of 12.7–19.0%.

The glasses considered were melted in an electric furnace with corundum heaters at a temperature of 1250°C in glass-carbon dishes for 40 min. Casting was performed into a cold mold; annealing was conducted at a temperature of 400–420°C. Thermal treatment after annealing was implemented according to a two-stage schedule: temperature rise

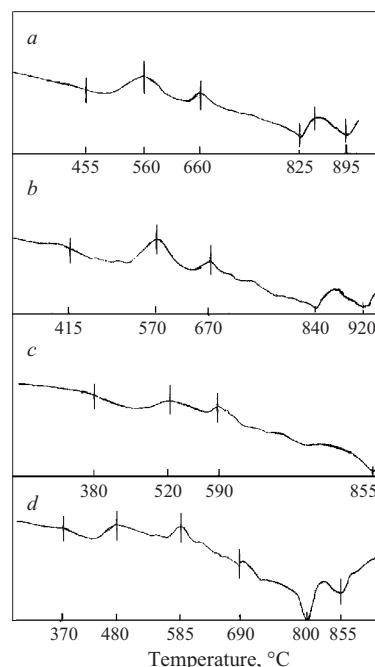


Fig. 1. Results of differential-thermal analysis of glasses 1 (*a*), 2 (*b*), 3 (*c*), and 4 (*d*).

TABLE 1

Composition*	Molar content, %						
	SiO ₂	Li ₂ O	K ₂ O	Al ₂ O ₃	CaF ₂	LiF	NaPO ₃
1	62.5	22.4	0.9	2.8	0.9	8.0	2.0
2	61.2	24.6	1.0	2.0	0.8	8.1	2.0
3	61.5	22.2	0.9	1.0	2.1	7.9	2.0
4	62.0	22.4	1.0	1.0	0.8	8.0	1.9

* CeO₂ content in all compositions amounted to 0.3%.

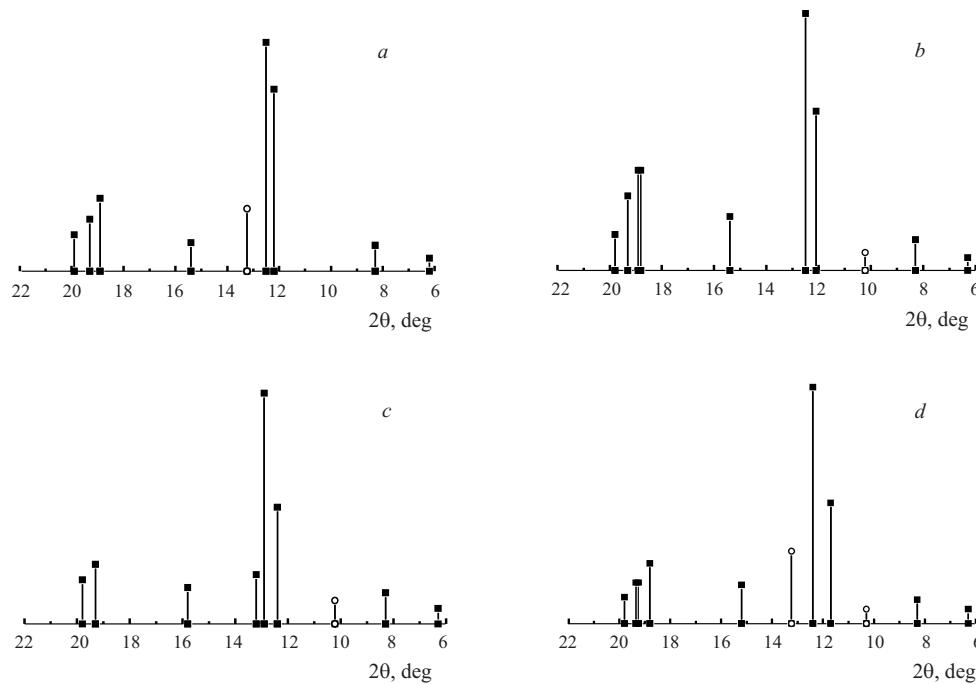


Fig. 2. X-ray diffraction patterns of glass-ceramic compositions 1 (a), 2 (b), 3 (c), and 4 (d): ■ lithium disilicate; ○ quartz.

up to 480–520°C at a rate of 3–5 K/min, exposure for 2–3 h, temperature rise up to 680–720°C at a rate of 1–4 K/min, exposure at the specified temperature for 1–2 h, and then natural cooling to room temperature.

The density of glass was measured by means of hydrostatic weighing in toluene, and the measurement error was $\pm 2 \times 10^{-3}$ g/cm³. Dilatometric curves were constructed using a DKV-4 vertical quartz dilatometer.

The density of glass ceramics obtained was 2.460–2.472 g/cm³, the TCLE was $(113 - 125) \times 10^{-7} \pm 1 \text{ K}^{-1}$.

The results of differential thermal analysis are shown in Fig. 1. The DTA curves recorded for our samples have some peculiarities common for all glasses of the lithium-aluminosilicate system prone to crystallization. As temperature increases, a descending segment emerges on the DTA curve caused by slight heat absorption due to the formation of crystallization seeds when temperature approaches the glass-annealing point.

According to DTA data, softening temperatures t_g in glass 1 is equal to 455°C, the exothermic effects are registered at 560 and 600°C, and the endothermic effects at 825 and 895°C. In glass 2, $t_g = 415^\circ\text{C}$, exothermic effects occur at 670 and 570°C and endothermic effects at 840 and 920°C. Glass 3 has $t_g = 380^\circ\text{C}$ and two exothermic effects at 520 and 590°C. Glass 4 has the lowest t_g equal to 370°C, exothermic effects are observed at 480 and 585°C, and endothermic effects at 800 and 855°C. The exothermic peaks related to heat release correspond to crystallization processes, whereas en-

dothermic effects (heat absorption) indicate the glass-melting processes.

X-ray phase analysis indicates that the main crystallization phases are lithium silicates and the silica phase (Fig. 2).

The static bending strength of glass ceramics of composition 1 is equal to 400 MPa.

The electric resistivity of glass ceramics is significantly higher than that of initial glasses and at 20°C is at the same level as that of the industrial insulator made of glass 13-V. At 150°C the resistivity of composition 1 is equal to: glass — $7.4 \times 10^7 \Omega \cdot \text{cm}$ and glass ceramic — $7.6 \times 10^9 \Omega \cdot \text{cm}$, for composition 2: glass — $8 \times 10^5 \Omega \cdot \text{cm}$ and glass ceramic — $3 \times 10^9 \Omega \cdot \text{cm}$, for composition 4: glass — $1.3 \times 10^7 \Omega \cdot \text{cm}$ and glass ceramic — $4 \times 10^9 \Omega \cdot \text{cm}$.

The hydrolytic resistance of glasses and glass ceramics obtained is classified as class I, except for glass ceramic 3 corresponding to class II.

The properties mentioned together with insignificant variations in the TCLE after glass crystallization makes these glass ceramics convenient for electric engineering. Since glass ceramics have low density and high mechanical strength, they are promising for service under extreme loads.

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